X-RAY DIFFRACTION MEASUREMENT OF STRESS RELAXATION ASSOCIATED WITH BUCKLING IN COMPRESSED THIN FILMS

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INTRODUCTION

Physical Vapor Deposition (PVD) techniques are widely used in in many technological areas such as microelectronics and bio medical applications (implants). Ion beam sputtering thin films bonded on non epitaxial substrates generally exhibit large compressive residual stresses due to the mismatch between the film and the substrate, and nanometric grain sizes. This compressive in-plane stress can spontaneously lead the film to buckle in order to relieve this stress; this phenomenon generally happen when the film deposited under vacuum is submitted to the atmospheric pressure outside the deposition chamber. Buckling patterns could have a simple shape like circular blisters or more complex ones like "Telephone Chords" wrinkles. These effects are undesirable and much effort was undertaken to theoretically study these phenomena by way of modeling and simulation [1].

The small dimensions of the buckling have so far prevented to obtain direct strain/stress measurements at the buckling location to support these theoretical results. The Micro-Raman technique may be applied for stress mapping at a micron-scale. However, this technique may only be considered for specific materials such as ceramics, oxides and semiconductors with well-defined microstructure and thus does not hold for metals. Furthermore, the Raman peak shift induced by stresses must be previously calibrated using stress-free reference samples which are sometimes difficult or impossible to obtain in the case of thin films. This calibration most often does not take into account the possible elastic anisotropy of the material. Hence, modification of the grain crystalline orientation in the film, which is related to elasticity when mapping strain field around a micro indentation or thin film buckling for examples, may lead to erroneous interpretation of the data.

EXPERIMENTAL METHOD

X-ray micro beams available on 3rd generation synchrotron radiations are now widely used in material science or for biological applications. Recent studies in cell biology, environmental science and microbiology using hard x-ray microprobes have yielded promising results [2] . In case of single crystals or when the x-ray beam is smaller than the crystallite size, coherent x-ray diffraction allows microstructure and strain/stress mapping measurements with submicrometre spatial resolution in three dimensions [3]. In case of nanocrystalline thin films (i.e. with nanometric grain size), this technique can not be applied because nanometric X-ray spot sizes are not yet available. However, due to rapid progress in x-ray optics [4], nanometric spot size would certainly be available in a new future for coherent x-ray diffraction analysis of strain/stress in nanostructures [5]. In this work, we apply a scanning x-ray microdiffraction (µSXRD) technique capable of resolving stress variations at micron level in nanocrystalline

samples. This technique, developed on the beam line 7.3.3.1 at the Advanced Light Source, take advantages of x-ray focusing optics at synchrotron sources of third generation, allowing to obtain micron to submicron size intense X-ray probes, large area Charge Coupled Device (CCD) technology and analytical methods for fast data collection and reduction with no required sample or detector rotation.

Investigated samples are 630 nm Au and 300 nm W polycrystalline thin films deposited at room temperature by ion beam sputtering technique on a 650 μm thick (100) Si wafer covered with native oxide. In the thin films, buckling are first identified using an optical microscope and their positions are precisely measured with respect to a visible reference position also identifiable by X-ray microfluorescence. We chose a step corner realized during the deposition process with a thin silicon mask. This allows to precisely locate the region of interest with the X-ray micro beam. The buckles are then scanned using a 10 to 15 μm step size and a typical 3 x 3 microns^2 x-ray spot size on the sample surface; at each step a diffraction pattern is collected with the CCD camera. A typical CCD diffraction pattern is displayed in Fig. 1(a). The grain size (< 10 nm) being typically much smaller than the beam size, the CCD frame yields a "powder" type rings. The intensity distributions on the rings are consistent with a preferential <111> fiber texture of the film. Residual stresses within the samples is determined by measuring the induced change in lattice constant with X-rays. The analysis of the diffraction patterns consists in applying the "d vs. $\sin^2\Psi$" technique to the rings, where ψ is the angle between the surface normal and the normal to the diffracting planes [6-7].
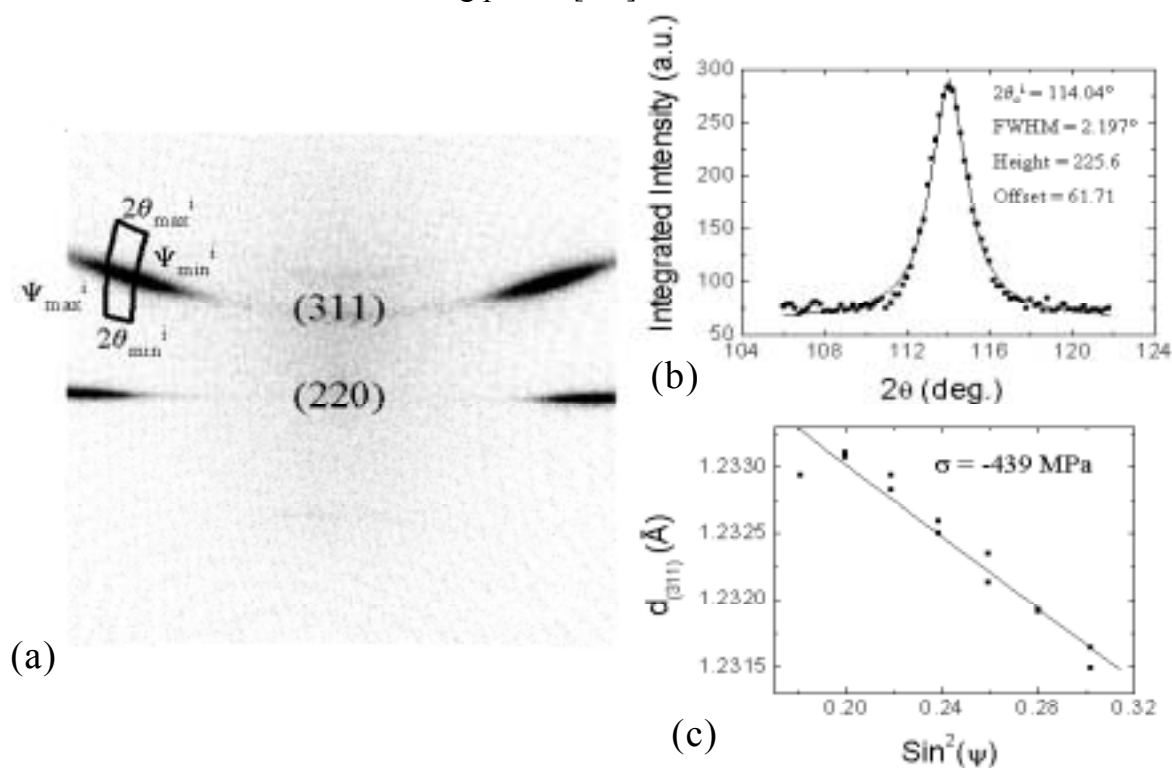

Figure 1.- *Principle of the $\sin^2\Psi$ technique applied to a 2D diffraction pattern. (a) Diffraction pattern obtained from a 630 nm thick Au film deposited on Si (λ = 0.2066 nm, exposure time: 300 s). The two visible rings have been indexed as (311) and (220). Intensity is integrated in a [ψ_{min}^i,ψ_{max}^i] delimited interval over a [$2\theta_{min}$,$2\theta_{max}$] range. (b) Result of the integration. The peak profile is fitted to a Lorentzian function and provides the central value $2\theta_c^i$ to be associated to the value ψ^i. (c) "d" vs. $\sin^2\Psi$ plot obtained after integration over 13 [Y^i] intervals.*
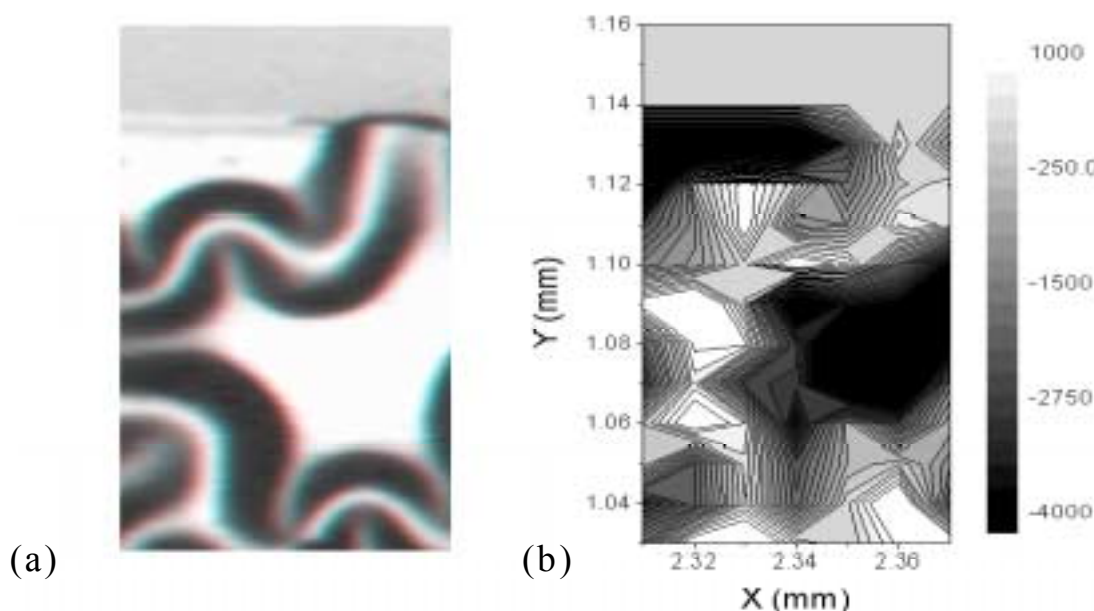
RESULTS

Figure 2.- *(a) Optical microscope image of wrinkled regions close to a step in a 300 nm thin W film deposited on Si. (b) Corresponding stress map obtained by µSXRD (λ = 0.2066 nm, exposure time: 300 s).*

Fig 2(b) shows the stress map as a result over a 60 µm x 140 µm area of buckling on a 300 nm thick W film on Si. The µSXRD scan was performed with a 10 µm step size. A nearly stress-free state is measured at the top of the buckling whereas adherent regions are strongly compressed. Topology of film buckling observed by optical microscopy and the measured X-ray stress maps are similar. More precise measurements are engaged as well as Finite Element calculations of the stress field associated with 2D blisters and wrinkles in these films. Confrontation of the stress profiles will permit to precise boundary conditions of the model and thus will lead to a better understanding of relevant parameters controlling thin film buckling.

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